REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
APPLICATION FOR A PATENT AND ACKNOWLEDGEMENT OF RECEIPT
(Section 30(1) Regulation 22)

FORM P.1
(to be lodged in duplicate)

REVENUE
-7. 3.91 R 130,00
INKOMSTE
REPUBLIEK VAN SUID AFRIKA
HASR 55

THE GRANT OF A PATENT IS HEREBY REQUESTED BY THE UNDERMENTIONED APPLICANT ON THE BASIS OF THE PRESENT APPLICATION FILED IN DUPLICATE.

PATENT APPLICATION NO.	
21 01	91 1688
71	FULL NAME(S) OF APPLICANT(S)

A & A REF: 122017 AS/CST

CSIR

ADDRESS(ES) OF APPLICANT(S)

Corporate Building, Scientia, PRETORIA, Transvaal Province,
Republic of South Africa

54 TITLE OF INVENTION

BINDER

Only the items marked with an "X" in the blocks below are applicable.

[X] THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2
[ ] THE APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO. 21 01
[ ] THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND BASED ON APPLICATION NO.
21 01

THIS APPLICATION IS ACCOMPANIED BY:

[X] 1. A single copy of a provisional or two copies of a complete specification of .... 15 ... pages
[ ] 2. Drawings of ..... sheets.
[X] 3. Publication particulars and abstract (Form P.8 in duplicate) (for complete only).
[ ] 4. A copy of Figure .... of the drawings (if any) for the abstract (for complete only).
[ ] 5. An assignment of invention.
[ ] 6. Certified priority document(s) (State quantity) : .....
[ ] 7. Translation of the priority document(s).
[ ] 8. An assignment of priority rights.
[X] 9. A copy of the Form P.2 and the specification of S.A. Patent Application No. 21 01 90/2226
[X] 10. A Form P.2 in duplicate.
[X] 11. A declaration and power of attorney on Form P.3.
[ ] 12. Request for ante-dating on Form P.4.
[ ] 13. Request for classification on Form P.9.
[X] 14. Request for delay of acceptance on Form P.4.
[ ] 15.

74 ADDRESS FOR SERVICE: Adams & Adams, Pretoria.

DATED THIS 7th DAY OF March 19 91

ADAMS & ADAMS
APPLICANTS PATENT ATTORNEYS

REGISTRAR OF PATENTS, DESIGNS, TRADE MARKS AND COPYRIGHT
RECEIVED
OFFICIAL DATE STAMP
1991 -03- 07
REGISTRAR OF PATENTS

The duplicate will be returned to the applicant's address for service as proof of lodging but is not valid unless endorsed with official stamp.

A & A P 201

ADAMS & ADAMS
SILVERBURG
PRETORIA

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
DECLARATION AND POWER OF ATTORNEY
(Section 30 - Regulation 8, 22(1)(c) and 33)

REPUBLIC OF SOUTH AFRICA REVENUE
7.3.91 R\001.00
INKOMSTE REPUBLIEK VAN SUID AFRIKA

FORM P.3

PATENT APPLICATION NO.	A & A REF: 122017 AS:CST	LODGING DATE
21 01 911688		22 7 March 1991

FULL NAME(S) OF APPLICANT(S)	
71	CSIR

FULL NAME(S) OF INVENTOR(S)	
72	GLADSTONE DAVIES SUSANNA VISSER

EARLIEST PRIORITY CLAIMED	COUNTRY	NUMBER	DATE
	33 ZA	31 90/2226	32 22 MARCH 1990

NOTE: The country must be indicated by its International Abbreviation - see schedule 4 of the Regulations

TITLE OF INVENTION	
54	BINDER

I/We ROY JOHN PAGE-SHIPP

hereby declare that :-

~~1. I/We am/are the applicant(s) mentioned above;~~

** 2. I/We have been authorized by the applicant(s) to make this declaration and have knowledge of the facts herein stated in the capacity of DIRECTOR; DIVISION OF BUILDING TECHNOLOGY of the applicant(s);

*** 3. the inventor(s) of the abovementioned invention is/are the person(s) named above and the applicant(s) has/have acquired the right to apply by virtue of the provisions of Section 13 of Act No. 46 of 1988;

4. to the best of my/our knowledge and belief, if a patent is granted on the application, there will be no lawful ground for the revocation of the patent;

**** ~~5. This is a convention application and the earliest application from which priority is claimed as set out above is the first application in a convention country in respect of the invention claimed in any of the claims; and~~

6. the partners and qualified staff of the firm of ADAMS & ADAMS, patent attorneys, are authorised, jointly and severally, with powers of substitution and revocation, to represent the applicant(s) in this application and to be the address for service of the applicant(s) while the application is pending and after a patent has been granted on the application.

DATED AT PRETORIA THIS 28 DAY OF FEBRUARY, 1991

SIGNATURE(S)
(no legalization necessary)

* In the case of application in the name of a company, partnership or firm, give full names of signatory/signatories, delete paragraph 1, and enter capacity of each signatory in paragraph 2.
** If the applicant is a natural person, delete paragraph 2.
*** If the right to apply is not by virtue of an assignment from the inventor(s), delete an assignment from the inventor(s) and give details of acquisition of right.
**** For non-convention applications, delete paragraph 5.

A. & A. Ref. No. 122017 AS/GST

ADAMS & ADAMS
PATENT ATTORNEYS
SHORBURG
PRETORIA

FORM P7

REPUBLIC OF SOUTH AFRICA
Patents Act, 1978

COMPLETE SPECIFICATION

(Section 30 (1) - Regulation 28)

OFFICIAL APPLICATION NO.	LODGING DATE
21 01 911688	22 7 March 1991

INTERNATIONAL CLASSIFICATION
51 C04B B28B B28C

FULL NAMES(S) OF APPLICANT(S)
71 CSIR

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72 GLADSTONE DAVIES SUSANNA VISSER

TITLE OF INVENTION
54 BINDER

THIS INVENTION relates to a binder. More particularly, it relates to a binder suitable for use as a substitute for ordinary portland cement.

According to one aspect of the invention there is provided a binder which comprises a Class F fly ash and an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, the binder being in the form of a particulate mixture comprising, for every 100 parts by mass of fly ash, 1 - 40 parts by mass of said alkali metal compound.

By Class F fly ash is meant fly ash classified by the American Society for Testing and Materials (ASTM) under Standard Specification Designation C618-87 as Class F fly ash. Typical chemical compositions of Class F fly ash available in South Africa are as set forth in the following table, Table 1:

TABLE 1

Constituent	% by Mass
SiO_2	40,04 - 50,6
Al_2O_3	26,30 - 38,93
Fe_2O_3	2,63 - 5,55
CaO	4,42 - 12,80
MgO	1,15 - 3,10
SO_3	0,16 - 1,13
Na_2O	0,30 - 1,03
K_2O	0,20 - 0,99
Loss upon Ignition	0,80 - 3,86

Optionally, the binder comprises a proportion, eg. 1-50 parts by mass of amorphous silica, for every 100 parts by mass of said fly ash. The amorphous silica may be fumed silica condensate or so-called silica fume.

Preferred proportions of the constituents depend to a degree on the nature of the constituents and the use for which the binder is intended. Thus, when the binder is intended for use in making cast products and the alkali metal compound is a sodium compound, the binder may contain 5 - 40 parts by mass of the sodium compound and up to 50 parts by mass of amorphous silica, for every 100 parts by mass of fly ash; when the binder is intended for use in making cast products and the alkali metal compound is a potassium compound, the binder may contain 7 - 40 parts by mass of the potassium compound and up to 50 parts by mass of amorphous silica, for every 100 parts by mass of fly ash; and when the binder is intended for use in making pressed products, it may contain 1 - 20 parts by mass of the alkali metal compound and up to 50 parts by mass of amorphous silica, for every 100 parts by mass of fly ash. For reasons of cost and availability, as indicated above, sodium and potassium alkali metal compounds are preferred.

More particularly, if the alkali metal compound is a hydroxide, then the preferred proportions depend on whether amorphous silica such as silica fume is added or not. Table 2 below indicates the preferred proportions of the different constituents on a parts by mass basis.

TABLE 2 (NO SILICA FUME)

Alkali Metal in Question	Fly Ash Proportion	Preferred Alkali Metal Hydroxide Proportion
Potassium	100	12 - 25
Sodium	100	6 - 25

Better strengths are obtained when silica fume is added to the mix with the best strengths obtained when the silica fume is first dissolved in the hydroxide solution made by mixing the required alkali metal hydroxide with the required amount of water before mixing in the fly ash. The preferred ranges of the different components are represented in Table 3 below.

TABLE 3 (AMORPHOUS SILICA ADDED)

Alkali Metal in Question	Fly Ash Proportion	Alkali Metal Hydroxide Proportion	Amorphous Silica (Fumed Silica Condensate) Proportion
Potassium	100	8 - 36	6 - 40
Sodium	100	5 - 30	10 - 50

It is also possible to use commercially available alkali metal silicate solutions or soluble powders, or mixtures of alkali metal silicates and alkali metal hydroxides, rather than mixtures of alkali metal hydroxides and amorphous silica. The preferred proportions for potassium and sodium silicates are presented in Table 4 below. It should be noted that the silicate proportions are presented on an anhydrous basis.

TABLE 4

Alkali Metal in Question	Fly Ash Proportion	Alkali Metal Silicate Proportion
Potassium	100	10 - 40
Sodium	100	19 - 40

The amount of water used in combination with the other constituents affects the strength of the binder. Generally, the higher the water content the weaker the strength of the

binder. However in the case of cast products a minimum water content is required in order for the mix to be workable and therefore to be compacted properly.

The fly ash and alkali metal compound may together form at least 66% by mass of the binder, preferably at least 80%.

Typically the binder will be used in a fashion similar to that of ordinary portland cement. Thus, it will be thoroughly mixed with water and optionally with a particulate filler or diluent such as granular particles (eg. sand, additional fly ash or granulated slag), fibres (eg. glass, steel, organic polymers or asbestos), and/or aggregate (gravel or stone), after which it is cast, pressed, moulded or extruded and then caused or allowed to set.

With regard to these fillers or diluents, the proportions thereof used will depend on what degree of shrinkage or expansion can be tolerated and/or the strength required in the eventual set product. For mortars and concretes the binder:filler/diluent (sand or stone) mass ratio may be 1:1 - 1:9 for continuously graded fillers or diluents; and as a first approximation proportions, particle sizes and/or particle size distributions will be employed which are similar to those employed in portland cement/concrete practice.

The invention extends to a method of making an artifact which method comprises admixing a binder as described above with water, shaping the mixture into a green artifact of a desired shape, and causing or allowing the green artifact to set, 8 - 200 parts by mass of water being admixed with the binder for every 100 parts by mass of the fly ash in the binder.

The shaping may be by casting, 20 - 200 parts by mass of water being admixed with the binder for every 100 parts by mass of fly ash in the binder, the alkali metal compound of the binder being selected from sodium compounds, potassium

compounds and mixtures thereof. Instead, the shaping may be by pressing, 8 - 20 parts by mass of water being admixed with the binder for every 100 parts by mass of the fly ash.

The green artifact may be caused to set by maintaining it at an elevated temperature of 40 - 100°C, in eg a conventional oven or a microwave oven for a period of a few minutes up to several, eg 4, days, with the artifact enclosed in a water-impermeable enclosure such as a plastics or similar water-impermeable membrane, or being located under water to prevent it from drying out prematurely. This curing at elevated temperature leads to enhanced early strength after 1 - 4 days. The green artifact can instead be allowed to set at an ambient temperature of not more than 40°C, while being maintained in a moist condition, but strength development thereof is somewhat slower.

As indicated above, the method may comprise admixing a particulate filler or diluent with the binder and water. The nature of the diluent or filler used and the proportions thereof will depend on the artifact to be made, examples being 8 - 20 parts by mass of asbestos fibre for every 100 parts binder for fibre-reinforced sheets; and 100 - 900 parts by mass of clean sand for every 100 parts binder for general purpose mortar applications. In general, concrete practice when employing portland cement can be used as a guide in this regard.

The invention also extends to a method of making an artifact which comprises admixing fly ash, an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, and water, shaping the mixture into a green artifact and causing or allowing the artifact to set, 1 - 40 parts by mass of said alkali metal compound being admixed with every 100 parts by mass of fly ash.

If the alkali metal compound and fly ash are not premixed into a binder as described above, the alkali metal compound may first be dissolved in the water to form a solution, after which the fly ash is admixed into the solution. If amorphous silica is used, it may be admixed with the solution before the fly ash, any other constituents being admixed into the solution after the fly ash.

The invention extends further to a binder which comprises fly ash and an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, the fly ash and alkali metal compound together making up at least 66% by mass of the binder, preferably at least 80%.

The invention extends still further to an artifact whenever made in accordance with the method described above.

The invention will now be described, by way of illustration, with reference to the following non-limiting Examples, in which the fly ash used was a Class F fly ash received from the Matla Power Station having the following chemical analysis:

Constituent	% by Mass
SiO_2	46,60
Al_2O_3	33,86
Fe_2O_3	4,22
CaO	9,31
MgO	2,20
SO_3	0,76
Na_2O	0,52
K_2O	0,20
Loss upon Ignition	1,54

The fly ash had a particle size such that 6% by mass thereof was retained on a test sieve of 0,045 mm nominal size, a specific surface of 3400 cm^2/g and a density of 2,31 (ie g/cm^3).

The sand used was clean quartz sand from Philippi, Cape Province, having a particle size such that it all passed through a test sieve of 0,850 mm nominal size and not more than 10% by mass thereof passed through a test sieve of 0,600 mm nominal size.

The fumed silica condensate used was obtained from a ferrosilicon plant in Pietersburg and had the following chemical analysis:

Constituent	% by Mass
SiO_2	94,06
Al_2O_3	0,72
Fe_2O_3	0,08
CaO	0,13
MgO	0,47
SO_3	0,42
Na_2O	0,23
K_2O	0,42
Loss upon Ignition	2,43

The fumed silica had a relative density of 2,27 and a specific surface of 37 000 cm^2/g.

The sodium hydroxide and potassium hydroxide used were of laboratory grade and were in the form of pellets.

SABS (South African Bureau of Standards) Method No. 866 was followed in the making of mortar specimens. Mixing was

effected by a mechanical mixer as described in SABS Method No. 866. The green mix was placed in steel prism moulds and compacted as described in SABS Method No. 866. After compaction the moulds were inserted into plastics bags having a volume of 10 litres which were then sealed to prevent moisture from escaping. Unless stated otherwise the green mix was cured at 40°C for a predetermined period, after which a cured or set artifact was obtained, whose strength was tested in conventional fashion using methods used to test cement or concrete artifacts. An Instron testing machine was used to determine the flexural strength of all the artifacts. For testing compressive strength said Instron machine was used except for artifacts with a compressive strength of over 40 MPa, in which case a Baldwin testing machine was used.

In all examples the proportions are given on a mass basis.

Example 1

A binder composition comprising 20,2 parts potassium hydroxide for every 100 parts fly ash was used together with 30 parts of water and 300 parts of sand. After two days curing the resulting artifact, which amounted to a mortar, had a compressive strength of 1,69 MPa, which increased after 9 days to 14,75 MPa, and after 26 days to 23,0 MPa. The equivalent flexural strengths were respectively 0,7 MPa, 4,1 MPa and 6,3 MPa.

Example 2

In this case, a binder composition comprising 28,21 parts potassium hydroxide and 15,09 parts amorphous silica (fumed silica condensate) for every 100 parts fly ash was used together with 38,4 parts water and 345 parts sand. In this case, after 2, 9 and 26 days respectively, a mortar artifact was obtained whose compressive strengths were 2,27 MPa, 22,66 MPa

and 25,0 MPa. The equivalent flexural strengths after the same periods were respectively 0,8 MPa, 4,6 MPa and 6,3 MPa.

Example 3

In this case a binder comprising 9,05 parts sodium hydroxide for every 100 parts fly ash was used together with 38,89 parts water and 300 parts sand. In this case after 2, 7 and 26 days respectively, a mortar artifact was obtained whose compressive strengths were 2,47 MPa, 24,34 MPa and 33,0 MPa, the equivalent flexural strengths after the same period respectively being 0,7 MPa, 4,5 MPa and 6,5 MPa.

Example 4

In this case the binder was not pre-mixed, but its various constituents were mixed separately to make up the green mix. For every 100 parts fly ash used, there were used 14,25 parts sodium hydroxide, 21,50 parts fumed silica condensate, 44 parts water and 360 parts sand. The sodium hydroxide was first dissolved in the water and then the fumed silica condensate was added to the solution and dissolved therein with mixing. The resulting silicate solution became very hot and was left to cool until it reached ambient temperature. The solution was then thoroughly blended and mixed with the remaining constituents to form the green mixture. In this case, after one day a mortar artifact was obtained which had a compressive strength of 11,6 MPa, which increased after 7 days to 50,9 MPa, and after 14 days to 64,2 MPa. After the same periods the respective equivalent flexural strengths were 2,3 MPa, 6,8 MPa and 8,6 MPa.

EXAMPLE 5

In this case, a binder composition comprising 8,19 parts sodium hydroxide and 11,62 parts amorphous silica (fumed silica condensate) for every 100 parts fly ash was used together with 30,21 parts water and 335 parts sand. The sodium hydroxide was first dissolved in water and then the fumed silica condensate was added to the solution and dissolved therein with mixing. The resulting silicate solution became very hot and was left to cool until it reached ambient temperature. The solution was then thoroughly blended with the fly ash and then the sand was added. In this case, after two days the mortar artifact obtained a compressive strength of 31,44 MPa, which increased after 7 days to 51.56 MPa and after 14 days to 51,63 MPa. After the same periods the respective equivalent flexural strengths were 5,05, 7,14 and 6,25 MPa respectively.

EXAMPLE 6

In this case, a binder composition comprising 12,22 parts sodium hydroxide and 22,93 parts amorphous silica (fumed silica condensate) for every 100 parts fly ash was used together with 43,47 parts water and 178,16 parts sand. The sodium hydroxide was first dissolved in water and then the fumed silica condensate was added to the solution and dissolved therein with mixing. The resulting silicate solution became very hot and was left to cool until it reached ambient temperature. The solution was then thoroughly blended with the fly ash and then the sand was added. In this case the specimens were cured at room temperature. The flexural and compressive strengths at different times are given below:

Time (Days)	Flexural Strength (MPa)	Comressive Strength (MPa)
1	0,9	2,1
2	1,8	4,3
7	3,5	16,8
14	4,9	28,0
21	6,9	37,7
28	6,7	35,8

EXAMPLE 7

The soluble sodium silicate used in this Example was obtained from Silicate and Chemical Industries, as so-called Silchem SP20 grade, with a reported SiO_2:Na_2O ratio of 2:1 and a mean total solids content of 80% by mass.

In this case, a binder composition comprising 30,19 parts sodium silicate for every 100 parts fly ash was used together with 45,33 parts water and 360,00 parts sand. The sodium silicate was first dissolved in water and the solution was then thoroughly blended with the fly ash and then the sand was added. In this case, after two days the mortar artifact obtained a compressive strength of 9,88 MPa, which increased after 7 days to 23,34 MPa, and after 26 days to 34,28 MPa. After the same periods the respective equivalent flexural strengths were 2,83, 4,45 and 5,67 MPa respectively.

1. A binder which comprises a Class F fly ash and an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, the binder being in the form of a particulate mixture comprising, for every 100 parts by mass of fly ash, 1 - 40 parts by mass of said alkali metal compound.

2. A binder as claimed in claim 1, which comprises 1 - 50 parts by mass of amorphous silica for every 100 parts by mass of said fly ash.

3. A binder as claimed in claim 1 or claim 2, in which the fly ash and alkali metal compound together form at least 66% by mass of the binder.

4. A method of making an artifact, which method comprises admixing a binder as claimed in claim 1 with water, shaping the mixture into a green artifact of a desired shape, and causing or allowing the green artifact to set, 8 - 200 parts by mass of water being admixed with the binder for every 100 parts by mass of fly ash in the binder..

5. A method as claimed in claim 4, in which the shaping is by casting, 20-200 parts by mass of water being admixed with the binder for every 100 parts by mass of the fly ash in the binder, the alkali metal compound of the binder being selected from sodium compounds, potassium compounds and mixtures thereof.

6. A method as claimed in claim 4, in which the shaping is by pressing, 8-20 parts by mass of water being admixed with the binder for every 100 parts by mass of the fly ash.

7. A method as claimed in any one of claims 4-6 inclusive, in which the green artifact is caused to set by maintaining it at an elevated temperature of 40-100°C with the artifact enclosed in a water-impermeable enclosure or located under water.

8. A method as claimed in any one of claims 4-7 inclusive which comprises admixing a particulate filler or diluent with the binder and water.

9. A method of making an artifact which comprises admixing fly ash, an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, and water, shaping the mixture into a green artifact and causing or allowing the artifact to set, 1-40 parts by mass of said alkali metal compound being admixed with every 100 parts by mass of fly ash.

10. A method as claimed in claim 9, in which the alkali metal compound is first dissolved in the water to form a solution, after which the fly ash is admixed into the solution.

11. A method as claimed in claim 10, in which amorphous silica is admixed into the solution before the fly ash, any other constituents being admixed into the solution after the fly ash.

12. A binder which comprises fly ash and an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, the fly ash and alkali metal compound together making up at least 66% by mass of the binder.

13. A binder, substantially as described herein.

14. A method of making an artifact, substantially as described herein.

DATED THIS 7TH DAY OF MARCH, 1991

ADAMS & ADAMS
APPLICANTS PATENT ATTORNEYS